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WAVEGUIDES BASED UPON CHALCOGENIDE GLASSES

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Because of their transparency in the infrared, their high refractive index and their well-known photosensitivity properties, interest in chalcogenide glasses for integrated optics and technological applications related to detection in the IR spectral domain has been growing during the past few years. Our present work is focused on the possibility of integrating chalcogenide glasses in components for two types of applications: spatial interferometry (detection of planets) and environmental metrology (detection of pollutant gases). In both cases, monomode channel or rib waveguides working in the infrared are to be obtained. In this context, first step of our work has been to optimize the chalcogenide films deposition conditions starting from glasses based on germanium combined with antimony and selenium, or with arsenic and selenium. Films with good adherence and controlled composition are currently obtained by RF-sputtering and thermal evaporation. Multimode planar chalcogenide waveguides, based on two external $Ge_{29}As_{12}Se_{59}$ and an inner $Ge_{21}Sb_{18}Se_{61}$ glassy layers deposited on glass or Si/SiO_2 substrates, were then fabricated and proved to guide at 1.55 μm. In order to realize channel or rib chalcogenide waveguides, Ar plasma etching and NH_4OH chemical etching of the films were also investigating and proved to be encouraging.

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1. Introduction

Interest in chalcogenide glasses for integrated optics has been growing during the past few years [1-5]. Most of the current research efforts deal with telecommunication applications, at 1.3 or 1.55 μm wavelengths, but chalcogenide glasses present wide transmission windows, from 1 to 20 microns depending on the material composition, high refractive indexes and numerous photoinduced properties (photodarkening, photodissolution of metals,...) which make them also competitive materials for several technological applications related to detection in the IR spectral domain. The present work is focused on two of these potential applications. The first one is spatial interferometry, which implies the use of components working in the far infrared for the detection and the spectroscopic study of extrasolar planets [6]. The second one is environmental metrology. Indeed, the detection of some vibrational modes present in some pollutant gases and located in the wavelengths between 3 and 12 μm would be possible thanks to optochemical sensors working in the infrared. In both cases, monomode channel or rib waveguides must be obtained. In our work, we preliminary focused on the possibility of fabricating multimode planar, channel or rib chalcogenide waveguides working in a large window of wavelengths (between 1 and 15 microns approximately). Adjusting the working wavelength will then only need adjustment of the size of the waveguide.

To date, our work has consisted in the choice of the chalcogenide glasses, the deposition of films with controlled characteristics and the realization of multimode planar waveguides in the continuity of our previous work [7]. The possibility of realizing monomode waveguides has also been investigated: the obtention of monomode structures implies the modification of the initial planar geometry of the films so chemical and plasma etching tests have been performed.

Progress in our investigation is presented in this paper.

2. Experimental

2.1 – Substrates

Two types of substrates were used: microscope slides and Si/SiO_2 substrates (1×1 cm^2) provided by the CNM (Centro de Microelectronica) in Barcelone and which oxidized layer thickness is about 40 nm.

2.2 – Film deposition

Films based on germanium combined with antimony and selenium were deposited by thermal evaporation of powdered $Ge_{28}Sb_{12}Se_{60}$ glass prepared by the conventional melt-quenched method, at a residual pressure of 2×10^{-3} Pa. The layer thickness was controlled by the quantity of powder in the molybdenum crucible (typically 250 mg for a thickness of 1 micron when the crucible is placed at 10 cm of the substrate). The deposition of the films based on germanium combined with arsenic and selenium was performed by RF-sputtering from a $Ge_{33}As_{12}Se_{55}$ commercial target (target called IG2, from VITRON AG), with a constant operating pressure of 5 Pa of argon for non-reactive sputtering. In order to prevent damage of the target, a 30 W RF sputtering power was used, yielding deposition rates of about 10 nm.min^{-1}.

2.4 – Film characterization

The adherence of the films on the substrates was verified by the classical adhesive tape test. Their thickness was estimated by profilometry (using a DEKTAK 3) but also by ellipsometric experiments carried out at the ICMAB (Institut de Ciencia dels Materials de Barcelona) in Barcelone and their surface aspect was inspected by Scanning Electron Microscopy (SEM) on a Leica Stereoscan 260. Chemical composition was estimated by Secondary Diffused Electron (EDS). Information about refractive indexes and absorption of the films was provided by ellipsometric measurements performed at the CNM in Barcelone in the range 0.7 to 5 eV.

The experimental setup used for the characterization of the waveguides is composed of a 1.55 micron source from Photonics coupled with a silica fiber which is positioned in front of the planar waveguide. The optical near field collected at the output of the waveguide is focused on a detector using a microscope objective.

Thickness and width of the ribs obtained by etching were measured by profilometry.

2.4 – Film etching

Two ways of etching were tested: (i) chemical (wet) and (ii) physical (dry) etchings.

In both cases, chalcogenide glass films were covered with a thin layer of Microposit® resin of about 2μm thickness by spin coating. After UV insolation through a mask (with 120 or 15 microns line motifs) and resin development in MF320 developer, the samples were annealed 30 minutes at 393 K.

(i) Chemical etching [8, 9]

Several alkaline solutions at different concentrations were tested and a 10^{-3} M NH_4OH solution was finally chosen. After treatment in this solution, layers were dipped in acetone to remove the non-irradiated resin regions.

(ii) Physical etching [10, 11]

After annealing at 393 K, films were introduced in the place of the target in the same sputtering chamber than the one used for depositing them. A 10 W Ar plasma was used (5 Pa Ar) and duration of the etching was chosen according to the required rib depth. After etching, layers were dipped in a commercial removal to remove the remaining resin regions.

3. Results and discussion

Two chalcogenide glasses were chosen for the realization of the future monomode waveguides: $Ge_{28}Sb_{12}Se_{60}$ and $Ge_{33}As_{12}Se_{55}$. Both glasses can be obtained commercially and present satisfying characteristics. They are characterized by high glass transition temperatures (551 K for the first one and 635 K for the second one), that is not negligible for their future utilization. They present a wide transmission window (1-14 microns for $Ge_{28}Sb_{12}Se_{60}$ and 0.8-16 microns for $Ge_{33}As_{12}Se_{55}$) : components based on these glasses could then work in the infrared but also at the telecommunication wavelength of 1.55 μm. These glasses are also characterized by high refraction index (2.6266 for the first one and 2.5469 for the second one at 1.55 μm).

The selected glasses were deposited in the form of thin layers and the deposition parameters were optimized. Good adherence of the films is now currently obtained, whatever the deposition method (RF-sputtering or thermal evaporation) and the substrate (microscope slide or Si/SiO_2) used. Characteristics of the films are also controlled. Two typical examples for films of each composition are given in Table 1.

Table 1. Typical characteristics for the two families of films : composition obtained by EDS, thicknesses, refractive indexes at two different wavelengths and absorption coefficients of two films given by ellipsometry.

Film	e (μm)	n (1.55 μm)	n (He-Ne)	k (He-Ne)
$Ge_{21}Sb_{18}Se_{61}$	1.2	2.78	3.40	~ 0.1
$Ge_{29}As_{12}Se_{59}$	1.9	2.25	2.35	~ 0

These characteristics are representative of all the deposited films. Some comments can be made about them. Film thicknesses obtained either by ellipsometry or by profilometry were proved to be similar. As expected for films deposited by thermal evaporation, composition of the "$Ge_{28}Sb_{12}Se_{60}$" films is quite different from the one of the bulk. The atomic percentage in germanium varies from 20 to 23 percents compared to the expected 28, the one in antimony from 16 to 19 instead of expected 12. The atomic percentage in selenium was proved to be less modified and constant at about 61 percents. Compositions of the "$Ge_{33}As_{12}Se_{55}$" films deposited by RF-sputtering are also somewhat different from the one of the bulk, but quite constant. Refraction indexes of the $Ge_{28}Sb_{12}Se_{60}$ and $Ge_{33}As_{12}Se_{55}$ bulk glasses are known to be 2.6266 and 2.5469 at 1.55 μm respectively. The difference between these values and the ones obtained for our films can be attributed to the difference in composition. To end, the precision in the absorption coefficients is poor (correct values can differ by a factor of 2) because the region in which they have been measured is very close to the absorption edge of the chalcogenide glasses.

Two multimode planar waveguides were then fabricated by stacking different films. They were both constituted of three layers, two external RF-sputtered $Ge_{29}As_{12}Se_{59}$ films constituting the clad and an inner thermal evaporated $Ge_{21}Sb_{18}Se_{61}$ film constituting the guiding core. Thickness of these layers was estimated by profilometry: the two external layers are characterized by a thickness of around 3.5 μm and the inner layer by a thickness of around 4.5 μm. In the waveguide called W_1, the layers were deposited on an oxidized silicon substrate, while in the one called W_2, they were deposited on a microscope slide substrate. After cleavage to obtain plane facets, these two waveguides were tested at 1.55 μm. To date, no guiding test has been performed at higher wavelengths because the sources were not available.

Fig. 1a shows the output of the waveguide W_1 through the inner $Ge_{21}Sb_{18}Se_{61}$ clad layer, and Fig. 1b shows the one of the waveguide W_2: a good guidance of the light is obtained in both cases. The guidance quality is better than the one obtained in a previous work [7]. As shown in Fig. 2, the output of the first planar waveguide made of a core $Ge_{12.5}Sb_{20}Se_{67.5}$ layer and an inferior $Ge_{28}Sb_{12}Se_{60}$ layer constituting the clad with air presented a granulous aspect. The latter was attributed to a damage in the layers during cleavage due to a lack of adherence of the layers on the substrate. On the contrary, the satisfying guidance obtained in the present work testify for a good adherence.

Fig. 1. Injection through the $Ge_{28}Sb_{12}Se_{60}$ core layer : (a) for W_1 ; (b) for W_2.

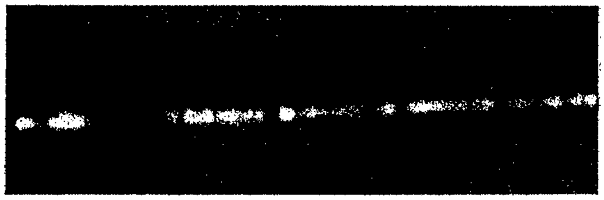

Fig. 2. Injection through the core layer of the waveguide described in reference [7].

Multimode planar chalcogenide waveguides are now currently obtained. The components required for spatial interferometry and environmental metrology applications are to be constituted of monomode waveguides, that is to say channel or rib structures (represented in Figs. 3a and 3b respectively). In both cases, modifying the initial planar geometry of the films is necessary. In this context, etching methods have been investigated.

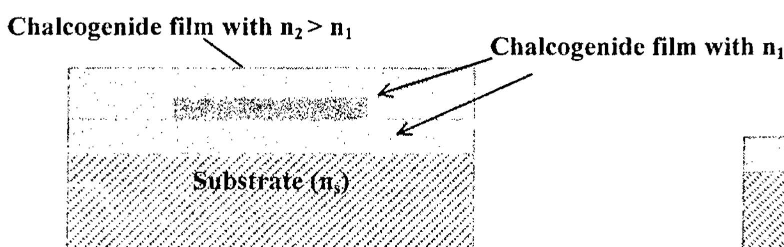

Fig. 3a. Representation of the "channel" structure to be obtained.

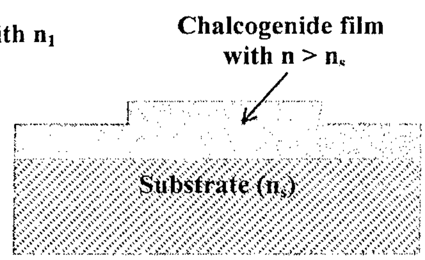

Fig. 3b. Representation of the "rib" structure to be obtained.

Chemical and physical etching tests were performed on $Ge_{29}As_{12}Se_{59}$ chalcogenide films deposited on Si/SiO_2 substrates.

These tests were proved to be very encouraging, since profiles of the as-obtained steps in the glassy films are good. As an illustration, SEM photograph of a physical etched film and profiles of characteristic steps obtained by chemical and physical etchings are presented in Figs. 5 and 6 respectively.

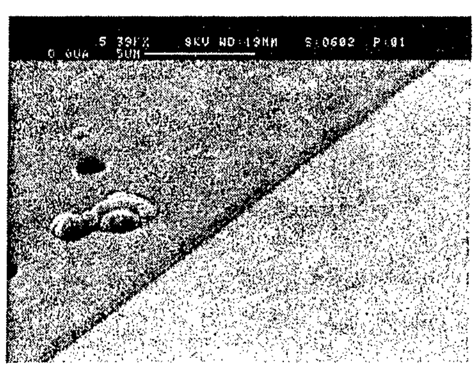

Fig. 5. SEM photograph of the ribs created by plasma etching.

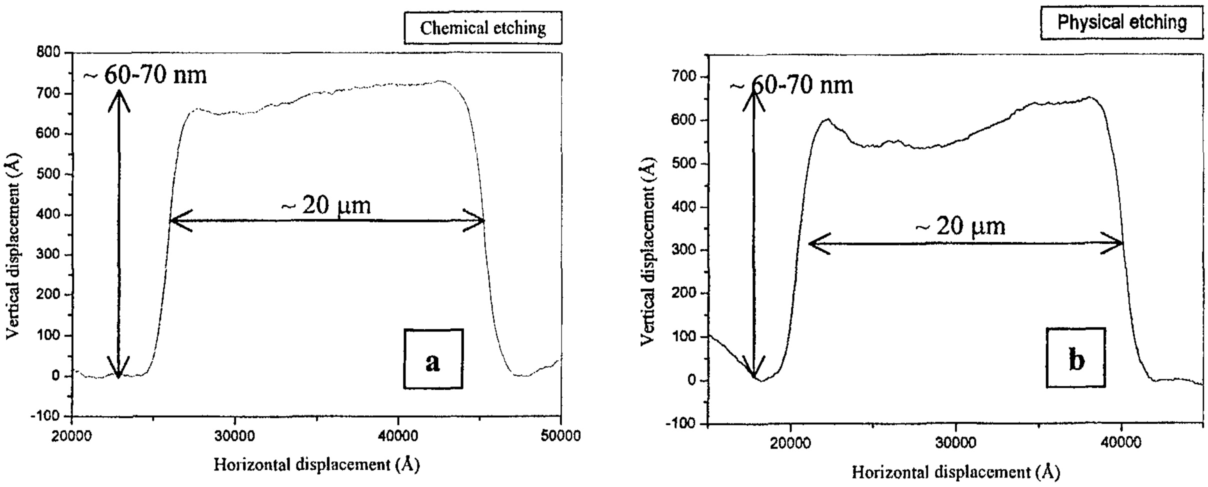

Fig. 6. Rib profiles obtained by profilometry for films: (a) chemically etched; (b) physically etched.

Up to now, tests have been performed with only one mask, so that the width of the steps is constant at 20 μm approximately. The depth of the steps was proved to be controllable and to date the smallest values obtained were 60-70 nm.

4. Conclusion

Films with good adherence and controlled composition are currently obtained, whatever the initial glass composition ($Ge_{33}As_{12}Se_{55}$ or $Ge_{28}Sb_{12}Se_{60}$), the deposition method (sputtering or thermal evaporation) and the substrate (oxidized silicon or microscope slide) are, for thicknesses up to 4 microns. Two multimode planar chalcogenide waveguides based upon $Ge_{29}As_{12}Se_{59}$ / $Ge_{21}Sb_{18}Se_{61}$ / $Ge_{29}As_{12}Se_{59}$ stacked layers were elaborated and proved to guide at 1.55 microns. Guiding quality was good but polishing the entrance surfaces rather than cleaving the substrates could still improve it.

First results related to the realization of channel or rib waveguides and the possibility of modifying the geometry of the films were very encouraging. Indeed, two etching methods (ie Ar plasma etching and NH_4OH chemical etching) were proved to be effective on $Ge_{29}As_{12}Se_{59}$ chalcogenide films.

Optimized dimensions of the waveguides to be suitable as far IR waveguides for astronomical interferometry or as optochemical sensors for environmental metrology are being calculated at the CNM in Barcelone. The next step will be the fabrication and characterization of such components with well defined geometry.

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